DETERMINATION OF THE COCOA BUTTER LIQUID SOAP SHAMPOO FORMULA USING RESPONSE SURFACE METHODOLOGY EXPERIMENTAL DESIGN

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ABSTRACT – *This study was conducted to formulate a liquid shampoo using cocoa butter liquid soap (CBLS). The CBLS has to be combined with other commercial surfactants to enhance the physical properties of the final product. The commercial surfactants selected as the factors were CBLS, cocamidopropyl betaine (COBET), decylglucoside (POLYG) and guar gum (GGUM). The final product, which was liquid shampoo in this study, was expected to have a pH of 7-8, a high foaming effect for dirt removal and good rheological properties. Response Surface Methodology (RSM) experimental design was used to determine the factors affecting the product parameter. The results indicated that the pH were significantly affected the factors. The foaming and rheological properties, however, were not significantly affected by the surfactant level in this study. The optimum pH value (pH 7.69±0.046) was obtained at the minimum amount of CBLS at 10 g, COBET at 28.1515 g, POLYG at 5.0215 g and GGUM at 0.4861 g, which was similar to the pH value (pH 7.6929) obtained using the model simulated. Subsequently, the product's rheological and foaming properties were reported in comparison with commercial products in the market.*

***Keywords*:** Cocoa butter liquid soap, surfactant, shampoo, product development.

INTRODUCTION

The haircare sector in Malaysia is forecast to grow from RM1,125.1M (US$269.2M) in 2019 to RM1,437.7M (US$341M) by 2024, recording a compound annual growth rate (CAGR) of 5.0% (Global Data, 2020). Further, the global hair care market is projected to grow at a CAGR of 3.35% during the forecast period (2021 - 2026), with the largest market is in Asia Pacific. The top three key players in the global hair market are Procter & Gamble, L'Oréal and Unilever; while in Malaysia, the sequence of leading companies are Unilever, L'Oreal SA and Procter & Gamble. The leading brands are Brylcream and Sunsilk, owned by Unilever. Male consumers accounted for a slightly higher share of 50.8% in 2019 when compared to female consumers who accounted for a 49.2% share, in the overall hair care sector in Malaysia. Since the trend showed promising business opportunities, the research and development on hair care products are worth to invest.

The word shampoo came from the Hindi word 'champoo' which means to press or massage; denoting cleaning through massage of the hair and skin (D'Souza and Rathi, 2015) and the modern shampoo contains synthetic surfactant which was first introduced in the 1930s. The main purpose of shampoo is to remove dirt and oil from the surface of the hair fibers and the scalp, without affecting the hair's internal keratin, lipid and filaments structure (Yang, *et al.*, 2014; Zhang, *et al.*, 2015). Ideally, shampoo can remove just enough sebum to clean hair and enough conditioning agents to leave the hair soft, shiny and manageable (D'Souza and Rathi, 2015).

Shampoos typically contain a primary and a secondary surfactant for thorough cleaning, a viscosity builder, a solvent, conditioning agents, a pH adjuster and other non-essential components such as fragrance and color for commercial appeal. Some shampoo comprises more than 10 ingredients (Trüeb, 2007). Nowadays, cosmetic manufacturers tend to use mild and non-irritant surfactants such as sodium cocoyl isethionate and cocamidopropyl betaine instead of sodium lauryl sulfate which is more economical. Although there is no scientific evidence on the carcinogenic effect of sodium lauryl sulfate, the misunderstood and misinterpretation by media and laypersons obscure the use of sodium lauryl sulfate as a cleansing agent in personal care products (Bondi, *et al*., 2015).

Cocoa has not been extensively studied to be used in hair care products. Cocoa butter was used in shampoo to moisturize and nourish hair without stripping as claimed by Palmer's® Cocoa Butter Formula® Moisture Rich Shampoo. Other examples of available shampoos containing cocoa materials in the market are Herbal Essences Whipped Cocoa Butter Shampoo, and Garnier Whole Blends Smoothing Shampoo with Coconut Oil and Cocoa Butter (**Error! Reference source not found.**). Cocoa materials, such as cocoa butter, have potential hair benefits such as moisturizing and hair protection. The formulation of hair shampoo with cocoa is a value-added to the

existing cocoa cosmetic personal care range being developed. In addition, the availability of these products in the market can increase the demand for cocoa.

Figure 1: Example of Available Shampoo containing Cocoa Butter

MATERIALS AND METHODS

Materials

The materials used in this study namely distilled water, cocoa butter liquid soap (CBLS; water (and) cocoa butter *Theobroma cacao* oil (and) *Ricinus Communis* seed oil (and) *Cocos Nucifera* oil (and) potassium hydroxide (and) glycerin (and) sodium chloride), glycerin, cocoamidopropylbetaine (COBET), decylglucoside (POLYG), glyceryl monostearate, hydroxypropyl trimonium chloride guar gum (GGUM), phenoxyethanol, pro-vitamin B5 and fragrance. All the chemicals and reagents were purchased from authorized chemical suppliers in Malaysia. In this study, Palmer's Cocoa Butter Formula Length Retention Shampoo (containing cocoa butter) and Pantene Daily Moisture Renewal Shampoo (commonly available in the shelf store) were selected for physical properties comparison with CBLS Shampoo. The ingredient function and lists of the shampoos are listed in Table 1.

Table 1: List of Ingredients & Functions of Commercial Shampoo

PRODUCT BRAND	Ingredients	Function
PANTENE DAILY MOISTURE RENEWAL SHAMPOO Intense moisturization with damage protection. Contain Pro-V Blends with rich lather to improve hair's ability to balance moisture  **320 mL (RM15.90)**	Water	Solvent
	Sodium laureth sulfate	Anionic surfactant
	Sodium lauryl sulfate	Anionic surfactant
	Dimethicone	helps to smooth hair, making it easier to brush and style without friction that can lead to breakage
	Glycol distearate	The pearlescent effect, moisturizing
	Fragrance	Product appearance
	Sodium citrate	pH adjuster
	Cocamide MEA	Cleansing agent
	Sodium xylenesulfonate	Anionic surfactant
	Cocamidopropyl betaine	Mild surfactant
	Sodium chloride	Thickening, viscosity adjuster
	Guar hyroxypropyltrimonium chloride	Hair conditioning agent
	Citric acid	pH adjuster
	Sodium benzoate	Preservative
	Tetrasodium EDTA	Chelating agent
	Histidine	Chelating agent
	Panthenol (vitamin B5)	helps with the shine, softness, and strength of your hair
	Panthenyl ethyl ether	Hair conditioning agent
	Oryza Sative (Rice) Bran Oil	Smooth and moisturize hair
	Methylchloroisothiazolinone	Preservative
	Methylisothiazolinone	Preservative
PALMER'S COCOA BUTTER FORMULA LENGTH RETENTION SHAMPOO system with Biotin, fully strengthens hair with powerhouse natural protectants that help block brittleness, breakage, and split ends to help hair achieve its optimal length,	Water	Solvent
	Theobroma Cacao (Cocoa) Extract	UV-damage protection
	Sodium C14-16 Olefin Sulfonate	Anionic Surfactant (pH 8-10)
	Glycerin	Skin-identical ingredients, humectant, moisturizer
	Glycol Distearate	The pearlescent effect, moisturizing
	Cocamide DIPA (diisopropylamine)	Non-ionic surfactant
	Cocoamidopropyl betaine	Mild surfactant
	Sodium chloride	Viscosity controlling
	PEG-120 Methyl Glucose Dioleate	Emulsifier
	PEG-150 Distearate	Thickener and emulsifier

Color safe; non-stripping removes build-up	Theobroma Cacao (Cocoa) Seed Butter	contains nourishing properties that shield the hair strand from the harmful effects of styling damage
 **400 mL (RM39.90)**	Biotin (vitamin B7)	helps to optimize the natural hair growth cycle; strengthens hair strands, preventing hair from becoming brittle and fragile reducing breakage
	Tocopheryl Acetate (Vitamin E)	hair is less prone to dullness and fading, leaving hair shiny and healthier
	Hydrolyzed Quinoa	Hair color retention, conditioning, and protection
	Cocos Nucifera (Coconut) Oil	Prevent protein loss from hair
	Leuconostoc/Radish Root Ferment Filtrate	Conditioning properties for hair
	Phenoxyethanol	Preservative
	Disteareth-75 IPDI (isophorone diisocyanate)	Emulsifier and thickener
	Polyquaternium-10	Conditioning agent, anti-static agent
	Sodium sulfate	Cleansing agent
	Ethylhexylglycerin	Preservative and skin conditioning agent
	Phosphoric acid	Help to reduce hair thinning or hair loss
	Tetrasodium glutamate diacetate	Stabilizer, prevent discoloration of the product, preservative booster
	Sodium glycolate	pH adjuster, skin conditioning agent
	Fragrance	Product appearance
	Benzyl benzoate	In fragrance; have the properties to fight insects and microbes

Methods

Process of Making CBLS Shampoo

The ingredient list of CBLS shampoo is listed in Table 2. The GGUM was added to the glycerin, then, dissolved into the boiling water and stirred vigorously until homogenous to form GGUM solution. In another beaker, the surfactant was heated to dissolve the glyceryl monostearate. The surfactant solution was added to the GGUM solution and mixed well. Phase C was added when the mixture has cool down.

Table 2: The ingredients list of CBLS shampoo

Phase	*Ingredients*	*Function*	*pH*
A	Distilled Water	Diluent	-
	Glycerin	Humectant	pH 7
	GGUM	Cationic surfactant	pH 9
B	CBLS	Anionic surfactant	pH 9-10
	COBET	Amphoteric surfactant	pH 5.50
	POLYG	Non-ionic surfactant	pH 11
C	Pro-vitamin B5	Additive	pH 8-9
	Phenoxyethanol	Preservative	pH 7
	Fragrance	Fragrance	-

The level of surfactant varied according to the list in Table 3, while the other ingredients remained throughout the study. The total number of experiments was 81 runs as designed by the Minitab software based on Box Behnken Design, alpha =1).

Table 3: The level of the factor for RSM experimental design

Factor	*Level*		
	Low	*Medium*	*High*
CBLS	10	20	30
COBET	10	20	30
POLYG	5	10	15
GGUM	0.1	0.5	1.0

pH Measurement

To measure the pH, 1 gram of the liquid shampoo was dissolved in 9 mL of distilled water. The measurement was conducted using a pH meter (Accumet, Fisher Scientific, U.S.A).

Rheological Measurement

The rheology of the liquid shampoo was measured using TA HR10 Rheometer (TA Instrument, U.S.A). The cone plate geometry was 40.0 mm, Peltier cone stainless steel plate. The data was collected and analyzed using the TRIOS Software. The infinite-rate and zero-rate viscosity was attained using the best-fit model of viscosity points (Pa.s) versus shear rate (1/s), while the yield stress and viscosity were retrieved by the best-fit model of viscosity (Pa.s) points versus shear stress (Pa).

Foam Ability & Stability

The foaming ability was determined using the cylinder shake method (Benjamin and Abbass, 2019; Gahlawat et al, 2019) with some modifications. To determine the foaming ability index, 1 g of the sample was put into a 100 mL graduated cylinder and the initial net height of the foam (Ho) was recorded before shaking for 30 seconds (H_1). Then, the net height of the foam after

leaving for 5 minutes (H_2) and after one hour was measured (H_3). The foam ability index was calculated as (H_2-H_0)/(H_1-H_0) and the foaming stability index was calculated as (H_3-H_0)/(H_2-H_0). The product with a foaming index value of more than 0.8 has good, 0.79-0.60 has moderate foaming and less than 0.60 has low foaming.

Accelerated Stability

The stability measurement was carried out using LUMiFuge 1113-81 (LUM GmbH, Germany). About 20 µL of the sample was inserted into the cuvette. The setting parameters for the measurement (300 profiles, 10 s intervals, 4000 rpm, light factor 1, at 25ºC) were predictions of about 3 months shelf-life.

Data Analysis

Minitab Software 16 (Minitab Pty Ltd, Australia) was used to evaluate the data. Response Surface Methodology was used as the experimental design. The factors were significantly affecting the properties measured when the p-value $\leq$ 0.05. The model was accepted when the R^2 is above 0.8. Consequently, one sample t-test was used to compare the simulation and experimental values. When the p-value $\geq$ 0.05, the model represents the data and can be used to predict the factor involved.

RESULTS AND DISCUSSIONS

In this study, we combined four types of surfactant to formulate the cocoa butter liquid shampoo. Cocoa butter liquid soap (CBLS) was produced by the saponification of cocoa butter, castor oil and coconut oil. The making of the CBLS was not discussed here.

The addition of GGUM, which is a water-soluble cationic polymer naturally derived from guar gum, acts as a non-gelling viscosity modifier, and its quaternary polymer structure provides conditioning and foam booster effects.

Provitamin B5 or D-panthenol was added to help moisturize the hair strands and helps to seal the moisture. D-panthenol forms a thin reflective coating on the surface of the hair for a shiny and vibrant look. It also has significant benefits to reduce hair loss (Shin, *et. al*., 2021).

Gavazzoni Dias, *et al*., (2014) mentioned that the alkaline pH of shampoo may increase the negative charge of the hair fiber surface, hence increasing friction between the hair, thus damaging the cuticle and breaking the hair fiber. Therefore, shampoo with low pH is recommended.

Each of the physical properties was analyzed, however, only pH was significantly affected by all the factors (Table 4). Consequently, the summary of the response surface regression for pH was in Table 5. The significance was given by the p-value $\leq$ 0.05 for each factor. CBLS and POLYG were the factors that influenced the increase of pH, significantly. On the other hand, COBET and GGUM reduced the pH value of the shampoo. In addition, the interaction between factors that most affected the pH of the shampoo was CBLS*POLYG (p-value of 0.000), followed by GGUM*POLYG (p-value of 0.003), COBET*POLYG (p-value of 0.008) and CBLS*COBET was significant at a p-value of 0.010.

Based on the results of response surface regression in Table 5, the model for the prediction of pH was represented by the following equation (1);

$$pH = 8.67842 + 0.03857(CBLS) - 0.05930(COBET) - 0.29611(GGUM) + 0.3617(POLYG) - 0.00196(CBLS * POLYG) + 0.02670(GGUM * POLYG) + 0.00106(COBET * POLYG) + 0.00051(CBLS * COBET) \quad \ldots\ldots\ldots (1)$$

The model is illustrated by the contour plot in Figure 2. To obtain shampoo with low pH (< pH 8), the formula should contain a low amount of CBLS with a high amount of COBET. Although shampoo with skin-friendly pH is preferred, the addition of cationic agents can help to minimize the irritation of the scalp and reduce frizz hair (Gavazzoni Dias, 2014).

The optimization for the factors to obtain low-pH shampoo was illustrated in Figure 3. The lowest pH that can be achieved by this model is pH 7.6929 by formulating the shampoo with the amount of CBLS at 10 g, GGUM at 0.4861g, COBET at 28.1515 g and POLYG at 5.0125 g. The origin pH of COBET has great influence to reduce the pH of the final product.

The said amount of surfactant was formulated in the laboratory and the value of pH7.693$\pm$0.0462 (

Table *6*) was gained similar to the pH value obtained by the model simulated (pH7.6929). Therefore, the model in equation (1) is valid to predict the pH of CBLS Shampoo within the range factors.

Table 4: Summary of Factor Levels and pH measured for RSM for CBLS Shampoo Formula

Run Order	CBLS	GGUM	COBET	POLYG	pH
1	20	0.1	20	5	8.39
2	10	1.0	20	15	8.87
3	20	0.1	20	10	8.59
4	20	1.0	30	15	8.43
5	10	0.5	10	15	9.14
6	20	0.5	20	15	8.72
7	20	1.0	20	5	8.40
8	30	0.1	20	5	8.78
9	10	1.0	30	15	8.24
10	10	0.5	30	5	7.63
11	20	0.1	10	5	9.17
12	30	1.0	30	15	8.67
13	30	0.1	20	15	8.95
14	30	1.0	30	10	8.68
15	30	1.0	10	5	9.12
16	10	0.5	20	5	7.80
17	10	0.1	30	10	7.99
18	20	0.5	20	5	8.55
19	20	0.1	10	10	9.08
20	10	1.0	10	15	8.90
21	20	0.1	10	15	9.05
22	30	0.1	30	5	8.47
23	10	0.1	10	5	8.51
24	20	0.5	30	10	8.37
25	10	0.5	10	5	8.44
26	10	0.1	10	10	8.80
27	10	1.0	20	10	8.13
28	10	0.5	10	10	8.71
29	20	0.5	10	10	8.96
30	10	0.1	30	15	8.14
31	30	1.0	20	5	8.75
32	20	0.5	10	15	9.05
33	10	0.1	20	10	8.39
34	30	1.0	20	10	8.97
35	10	1.0	30	10	7.93
36	30	0.1	30	15	8.60
37	30	0.5	10	10	9.20
38	10	0.1	20	5	8.55
39	30	1.0	10	10	9.29
40	10	0.5	20	10	8.26
41	30	0.5	10	15	9.36
42	30	1.0	10	15	9.42
43	30	0.5	10	5	9.13
44	20	1.0	20	15	8.87
45	30	0.5	20	10	8.85
46	10	0.1	20	15	8.57
47	30	0.1	10	10	9.20
48	10	0.1	10	15	8.86
49	30	1.0	20	15	9.00
50	20	1.0	10	10	9.12
51	20	0.5	30	5	8.06
52	30	0.5	30	5	8.44
53	10	0.5	30	15	8.15
54	20	0.5	20	10	8.62
55	30	1.0	30	5	8.41
56	20	0.1	30	10	8.28
57	20	0.1	30	15	8.60
58	20	1.0	10	5	8.88
59	20	1.0	20	10	8.65
60	20	1.0	10	15	9.09
61	30	0.1	10	5	9.34
62	20	1.0	30	5	8.02
63	20	0.5	30	15	8.43
64	20	1.0	30	10	8.39
65	20	0.5	10	5	9.14
66	20	0.1	20	15	8.88
67	10	0.5	20	15	8.59
68	10	1.0	10	10	8.85
69	30	0.1	10	15	9.26
70	30	0.5	20	5	8.95
71	10	1.0	10	5	8.57
72	30	0.5	30	10	8.77
73	30	0.5	30	15	8.62
74	30	0.5	20	15	8.99
75	10	1.0	30	5	7.26
76	30	0.1	20	10	8.85
77	10	1.0	20	5	8.06
78	20	0.1	30	5	8.21
79	10	0.5	30	10	7.87
80	30	0.1	30	10	8.57
81	10	0.1	30	5	7.64

Table 5: Response Surface Regression for pH versus CBLS, GGUM, COBET and POLYG

Estimated Regression Coefficients for pH

Term	Coef	SE Coef	T	P
Constant	8.67842	0.153071	56.696	0.000
CBLS	0.03857	0.005679	6.793	0.000
GGUM	-0.29611	0.092409	-3.204	0.002
COBET	-0.05930	0.005679	-10.442	0.000
POLYG	0.03617	0.012240	2.955	0.004
CBLS*COBET	0.00051	0.000193	2.650	0.010
CBLS*POLYG	-0.00196	0.000386	-5.069	0.000
GGUM*POLYG	0.02670	0.008555	3.121	0.003
COBET*POLYG	0.00106	0.000386	2.751	0.008

S = 0.115736 PRESS = 1.23881
R-Sq = 93.85% R-Sq(pred) = 92.10% R-Sq(adj) = 93.16%

Analysis of Variance for pH

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	8	14.7128	14.7128	1.83910	137.30	0.000
Linear	4	14.0427	4.4528	1.11321	83.11	0.000
CBLS	1	4.6171	0.6181	0.61808	46.14	0.000
GGUM	1	0.0093	0.1375	0.13754	10.27	0.002
COBET	1	7.9888	1.4605	1.46051	109.04	0.000
POLYG	1	1.4276	0.1169	0.11695	8.73	0.004
Interaction	4	0.6700	0.6700	0.16751	12.51	0.000
CBLS*COBET	1	0.0940	0.0940	0.09404	7.02	0.010
CBLS*POLYG	1	0.3442	0.3442	0.34418	25.70	0.000
GGUM*POLYG	1	0.1305	0.1305	0.13049	9.74	0.003
COBET*POLYG	1	0.1013	0.1013	0.10134	7.57	0.008
Residual Error	72	0.9644	0.9644	0.01339		
Total	80	15.6772				

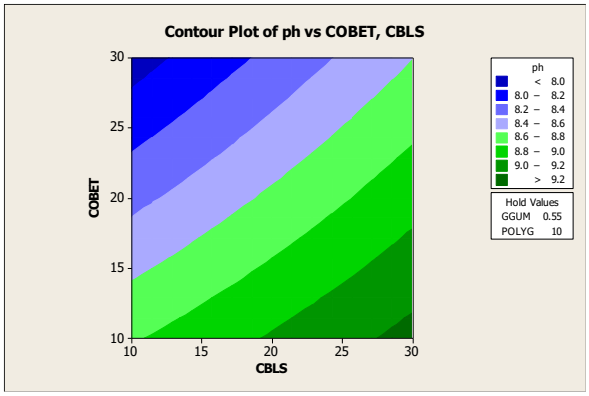

Figure 2: Contour Plot of pH by COBET and CBLS

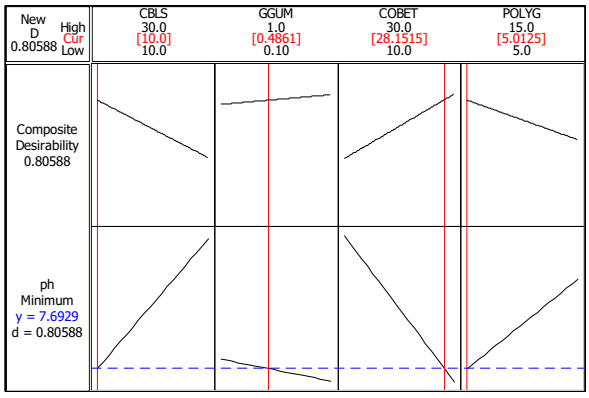

Figure 3: The Optimization Plot for pH versus CBLS, GGUM, COBET and POLYG

Table 6: Physical Parameter of CBLS Shampoo in Comparison with the Commercial Shampoo

Parameter	*CBLS Shampoo*	*Palmer's*	*Pantene*
pH	7.693±0.0462	7.19	5.815±0.005
Foam Ability	0.967±0.007	0.909±0.029	0.838±0.005
Foaming stability	0.943±0.049	0.867±0.140	0.913±0.788
Viscosity (Pa.s)	22.4715	123.275	98382.5
Yield Stress (MPa)	-3.37948e-5	-2.05172e-4	-0.0983438
Zero-rate Viscosity	86.3674	14.9894	9.8734
Infinite-rate Viscosity	-0.978743	-411.381	0.112141
Stability (3 months)	Stable	Stable	Stable

The CBLS Shampoo has very good foam ability and stability which was comparable to the commercial product (

Table *6*). Pantene was the most viscous shampoo compared to Palmers' and CBLS Shampoo based on the viscosity obtained. The viscous product usually has high yield stress indicating that high energy is needed to initiate the pumping process during manufacturing and when expelling the product during usage (Cook, 2019).

CBLS Shampoo also exhibited a higher zero-rate viscosity value compared to the other products which is an indication of a more stable product. Based on the infinite-rate value, CBLS has medium spreadability compared to the commercial brand. In addition, smaller gap values of infinite-rate and zero-rate viscosity exhibited that CBLS Shampoo and Pantene could easily get into the hairline in comparison with the Palmer's.

Figure 4 showed that all the shampoos were the shear-thinning non-Newtonian fluid. The products stayed in liquid form at stationary which is indicated by viscosity plateaus as the shear rate approaches zero (Duffy, 2016).

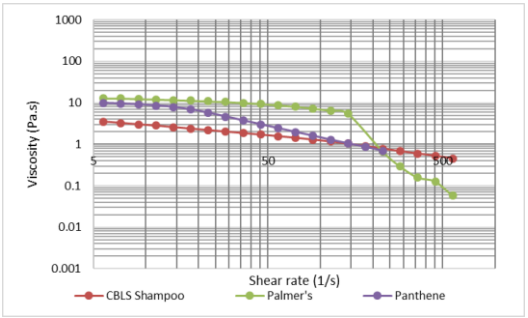

Figure 4: Viscosity vs Shear Rate of CBLS Shampoo in Comparison with Commercial Shampoo

Figure 5 showed the transmission profiles of CBLS Shampoo which represents the stability for (months). Since the transmission lines were on top of each other, therefore the CBLS Shampoo formula is stable for at least 3 months.

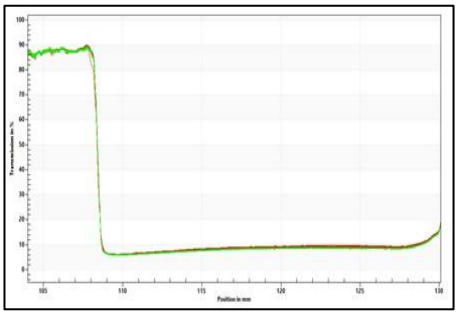

Figure 5: Transmission Profiles of CBLS Shampoo

CONCLUSIONS

In conclusion, the formula of CBLS Shampoo was affected significantly by the amount of the combination surfactant selected in this study where the pH was the main measuring parameter. The CBLS Shampoo was comparable in terms of physical properties to the available shampoo product in the market.

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